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Mechanical Properties of Solid Coatings

Joseph V. Koleske
Charleston, USA

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Coatings can be liquid or solid materials; they have been known and used from the time of early man and are of major importance for protecting and decorating the myriad of items in use today. These thin protective films are used to coat commercial and residential buildings (architectural coatings) and for many products in use today (industrial coatings). This article is concerned with the formation of coating films and the properties of these films. Static and dynamic mechanical properties, flexibility, toughness, adhesion, hardness, abrasion resistance, slip, chemical resistance, and stress within coatings are discussed and ways to investigate these characteristics are given. Some of the end-uses and the relationship of coating properties to the uses are detailed.

1 INTRODUCTION

The term “coating or coatings” is used to designate liquid or solid materials. A product that is to be applied to a substrate in a continuous or discontinuous film by one

of many application methods is a liquid coating. After the liquid coating is dried by evaporative means or is cured (cross-linked) by oxidative, thermal, ultraviolet light or other method it is a solid coating film. This article will deal for the most part with solid coating films and the properties of liquid coating films can be found elsewhere in the encyclopedia. Coatings can be colorless or colored; they are thin, solid films that are transparent, translucent, or opaque in nature. The terms “coatings” and “paint” should be synonymous, but often coatings refer to industrial coatings such as those used on appliances, office furniture, paper, automobiles, beverage cans, etc. and paint refers to architectural coatings such as house paints, wall and ceiling paints, trim paints, etc. The materials used by artists⁽¹⁾ are referred to as artists’ paints. While this is not a clear distinction, it is useful to be aware of a general differentiation in the terms and of the way they are used on a daily basis. Consider also that coatings are often referred to as finishes.

Paints can be defined as dispersions of pigments, optionally including fillers, in a fluid vehicle. The fluid vehicle comprises a liquid binder that will solidify during cure and, if necessary, a liquid carrier that serves as a viscosity reducing aid and will provide desirable application characteristics. The liquid carrier is lost to the local environment or recovered during the drying or curing process. The binder portion of the fluid vehicle is an oil, dissolved polymer, and, when appropriate, a cross-linking agent, or a dispersed polymer in latex or other dispersion form. The binder holds the pigment, filler particles, and miscellaneous formulating ingredients when it solidifies into a film on exposure to air or some other curing media. Varnish, a term that is often replaced by the term clear coating, is a clear or transparent solution that solidifies into a functional film. Lacquers are opaque and/or colored varnishes. Collectively or individually, paints, varnishes, and lacquers are termed coatings.

Coatings can be functional and/or decorative in nature. One need only to look at almost any surroundings to see how widely coatings are used. Many things that can be seen are coated with a decorative and/or a functional material. For example, within an office there might be walls and ceiling coated with a colored decorative paint; a desk coated with a clear, functional finish, which may be applied over a colored, decorative stain coating; alternatively the desk could be coated with a colored, opaque, functional and decorative coating; a floor coated in a manner similar to that of the desk with a functional and decorative coating; there may also be an aluminum beverage can coated on the outside with a decorative coating that serves as an advertising and identification medium and coated on the inside with a functional coating that protects the metal from the chemical nature of the can’s contents. In addition to the above, there may be

many books, perhaps a newspaper, and other printed material. Some of these are printed on coated paper, and the printing in itself is a coating that is discontinuous in nature. The book jackets and some of the illustrations in the books are continuous-film coatings. Sheets of labels found in an office are coated with a pressure-sensitive adhesive. The list could go on, but it is readily apparent that we are surrounded by coatings.

Decorative coatings can be brilliant and bright to attract visual senses quickly; pastel and soft to provide a restful atmosphere; single color or multicolor to provide a variety of dramatic effects; glossy, semi-glossy, or matt to provide different mood effects or to affect cleanability; rough or smooth, and so on. Functional coatings often protect substrates – wood, metal, plastic, or other – from the ravages of nature, which often provide a hostile environment and can cause wear through rusting, erosion, light attack, etc. The protection provided by functional coatings saves natural resources and is friendly to the environment because it minimizes corrosion and other means of degradation, thus allowing a substrate to last for a much longer period than it would without protection. Such coatings could also protect an expensive part of an assembly that cannot be reached or is difficult to reach for repair, such as the role conformal coating plays in the electronics industry. Here the coatings protect printed circuit assemblies from the hostile environment found in outer space and sometimes within plants, laboratories, or living quarters. Pressure-sensitive adhesives applied to the back of heavily coated paper or polyester film form widely used functional label products of various designs.

Coatings can be divided into two broad groups: architectural coatings and industrial coatings. Architectural coatings are those used in decorating and protecting houses and other buildings. If the coatings are used on the portion of the building exposed to atmospheric conditions, they are termed exterior coatings. Such coatings are usually made from materials that are not light (radiation) sensitive or are stabilized against attack by radiation of different wavelengths. If they are used on the inner portions of the buildings, they are referred to as interior coatings; this group is further broken down into ceiling paints, wall paints, varnishes, masonry paints, and stains.

2 HISTORY

Early humans used plant extracts, tree saps, animal fats, berry juices, and metal oxides to create paints that were used to decorate and communicate by means of pictures, often on cave walls.^(2,3) Such pictures have remained well defined and vibrant for over 15 000 years. Early Egyptians coated dead bodies with bitumen and other materials in

the mummification process (the word mummy is derived from the Persian word *mumia*, which means bitumen or pitch)^(3,4) and it is said that Noah's ark was coated with pitch. About 2500 years ago, Egyptians developed clear varnishes by heating amber and vegetable oils; colored coatings or lacquers were made by adding ground minerals such as malachite, azurite, and iron compounds to the clear varnishes. Early Romans developed the method of fresco painting in which paints composed of pigments, fillers, and carrier vehicles were applied to wet plaster for interior surface coatings and paintings. Ancient Hebrews used milk-based paints for decorating walls and ceilings. Over 3000 years ago, the Japanese developed lacquers based on sap from the varnish tree (the Japanese sumac, *Rhus verniciflua*). In colonial days, water-slacked lime – whitewash – was extensively used for a variety of coating purposes.⁽⁵⁾ Later whitewash was modified with milk and protein-based materials to improve durability and adhesion. This was followed by the addition of pigments, fillers such as clay, and whiting to provide a variety of products with improved aesthetic appeal and economics. Finally, the lime was replaced with milk phosphoproteins, and whitewash became casein paint, a forerunner of today's architectural paints.

Although paints were used and modified in such ways for centuries, it was not until the Industrial Revolution (1700–1950) that the paint and coating industry took on far-reaching importance. This historical period resulted in the production of a multitude of bridges, factories, manufacturing machinery, and allied equipment, as well as other items; all of these needed to be coated to provide protection from hostile natural and derived environments. Protection had to be provided from moisture, salt water, barnacles, mildew, mold, wind, rain, hail, heat and cold, sunlight, sulfurous fumes from coal fired furnaces, etc. This protection was provided by coatings that extended the lifetime of the world's infrastructure, the manufacturing facilities, and the ever increasing number of manufactured items. Today, the same items plus a myriad of other items – packaging, cars, trucks, trains, boats, furniture, beverage can liners, wallpaper, etc. – that grew out of the ever expanding manufacturing base are coated with a protective and very often aesthetically pleasing film that is usually thinner than a sheet of writing paper. If it were not for paint and coatings, our world would certainly be a dull, corroding place in which to live.

3 ARCHITECTURAL COATINGS

Architectural coatings are those coatings used on interior and/or exterior surfaces such as those found in or

on commercial, institutional, industrial, and residential buildings as well as on various structures such as bridges.⁽⁶⁾ The surfaces coated are wood, metal, composition, plaster and wallboard, plastic, or masonry. The coatings may be latex, alkyd, oil, solvent borne, and so on, and they are applied by brushing, spraying, rolling, as well as other methods. Gloss is an important optical characteristic of these coatings and varies from low to high depending on any particular end-use. These coatings are often termed trade sales paints, because they are usually purchased by consumers who will apply them on-site under ambient conditions.

Standard methods are available to test the characteristics of architectural coatings.⁽⁷⁻¹⁰⁾ Abrasion, dry and wet adhesion, flexibility, chemical resistance, block resistance, print resistance, and cleanability are important properties.

Abrasion resistance, adhesion, and flexibility are discussed elsewhere in this article. Mechanical properties of architectural coatings are not usually directly measured, but rather such properties manifest themselves in the results of an end-use-related test. Testing of architectural coatings and industrial coatings is often a measure of a complex interaction of various coating physical characteristics. For example, washability and related characteristics⁽¹¹⁻¹⁴⁾ of a wall coating requires the coating to be chemically resistant to water and detergent and to be sufficiently strong to withstand a scrubbing action that applies tensile and shear stresses to the coating as its hardness, adhesion, cohesion, and abrasion resistance are brought into play.

Ease of application and aesthetic characteristics are more important than mechanical characteristics for interior coatings. However, mechanical and other physical properties are important to exterior architectural coatings that are subjected to outdoor exposure. Exterior coatings are exposed to heat and associated temperature changes, moisture, oxygen, and sunlight. These factors individually or in concert contribute to coating failure. Temperature changes alter properties and can result in significant alternating strains, along with concomitant stresses,⁽¹⁵⁾ being placed on the coating-substrate matrix. This, coupled with the effects of exposure to relatively high temperatures, results in cracking, checking, embrittlement, and peeling. Moisture can cause blistering, erosion, loss of adhesion, and mildew. Oxygen can cause surface degradation and eventually internal degradation, resulting in embrittlement, cracking, and crazing. Sunlight and, in particular, the ultraviolet light component of sunlight can cause surface chalking and loss of gloss, degradation, and embrittlement, with accompanying cracking and discoloration.

Effects of outdoor exposure are often measured with test devices that attempt to simulate and accelerate

changes that would be encountered in the environment.⁽¹⁶⁾ Accelerated exposure test results are often difficult to reproduce and may not correlate with actual or natural exposure testing. However, such testing is widely used because natural weathering can take years to effect changes, and the accelerated tests do give a good indication of coatings that will fail early. They are particularly important for comparison purposes, for new product development, and to improve the durability of existing products.

Natural weathering⁽¹⁷⁾ is a true measure of the ravages of nature; however, it is reproducible only if properly planned, conducted, inspected, and reported. Natural weathering tests are not carried out by merely placing a test specimen outdoors and letting sunlight, rain, and so on fall on it. Rather, the testing is carried out at selected sites with the exposed specimens set at a particular angle. Conditions such as these affect the four major factors listed above and allow other factors such as humidity or lack of it, biodegradation, and pollution to be brought into the testing scheme. The synergistic interaction of these factors, which will vary with exposure site, determines how a coating's failure through outdoor exposure takes place. The variation of solar energy radiation dosage with season and with the angle of the test specimen to the sun is an important variable to be considered when reproducible test results are expected. The closer the test conditions simulate the actual use conditions, the more accurate will be the prediction of long-term results.

4 INDUSTRIAL COATINGS

Industrial coatings are coatings applied to factory-manufactured products. These include, but are not limited to, transportation coatings (those coatings applied to aircraft, appliances, automobiles, buses, recreational vehicles, trucks, and trains) beverage-can and spray-can coatings, packaging items, business machine and office furniture coatings, wood cabinet and furniture coatings, pipeline coatings, printed circuit board and assembly coatings, sign coatings, marine coatings, and masonry coatings.

4.1 Film Formation

Coating films are formed from either thermoplastic (soluble) or thermoset (insoluble) polymers combined with other ingredients, including, if desired, but not limited to, pigments, fillers, colorants, plasticizers, surfactants, solvents, catalysts, or initiators.⁽¹⁸⁻²⁰⁾ Thermoset coatings are formed from initially soluble ingredients that react and undergo a change termed cross-linking as a consequence of an energy input.

When thermoplastic polymers are dissolved in a solvent, films are formed by evaporation of the solvent under ambient conditions or in the presence of controlled heating. Since physical characteristics of the final coating are dependent on the polymer's properties, polymers of high or relatively high molecular weight are used. The high molecular weight limits the amount of polymer that can be dissolved because the viscosity of the final system must be sufficiently low to allow the coating to be applied by brush, spray, roll-coater, etc. Polymers that are used to form coating films include nitrocellulose, cellulose acetate butyrate, vinyl chloride/vinyl acetate copolymers and terpolymers, poly(vinyl acetate), and poly(methyl methacrylate). Coatings such as these are easily removable with a solvent or marred by a plasticizing compound; in addition particular liquids can attack the coating, for example water or alcohol can cause a ring on nitrocellulose-coated furniture.

Thermoset coatings are produced when multifunctional low-molecular-weight polymers or oligomers are reacted with a multifunctional cross-linking compound that contains appropriate functionality. If a solvent is present, it is removed by evaporation to form a film, which is then heated to effect reaction between the different functionalities in the presence of (or without) a catalyst. If the cross-linking reaction is between hydroxyl-containing and isocyanato-containing or epoxide-containing compounds, for example, the reaction proceeds by an addition process without emissions to form either urethane linkages or ether linkages (Scheme 1).

If the reaction is between a hydroxyl-containing compound and, for example, a methoxymelamine, material is lost by emission of a volatile by-product, in this case methanol (Scheme 2).

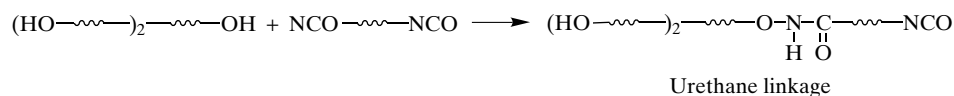
In addition to forming films by evaporation of solvent from solutions of polymers and oligomers, films can be formed from aqueous and non-aqueous dispersions, organosols, plastisols, electrodeposition, powders, and radiation-activated systems. The American Society for Testing and Materials (ASTM) and other organizations have a variety of test methods for determining various aspects of film formation and the films formed. These include detailed procedures for preparing⁽²¹⁾ and testing⁽²²⁾ organic films, for drying, curing, and formation

of films,⁽²³⁾ for determination of minimum film formation and coalescence temperatures of aqueous dispersions,⁽²⁴⁾ for permeability,⁽²⁵⁾ for block resistance,⁽²⁶⁾ and for numerous other particular physical factors. A number of the physical property tests are briefly described below.

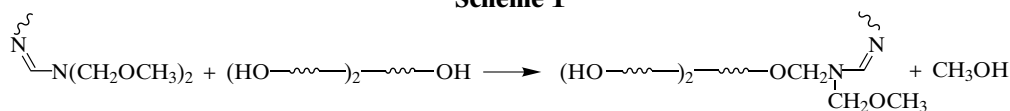
The degree of cure or solvent resistance of films, particularly thermoset coatings, is often determined by means of a solvent rubbing procedure that is formalized for zinc-rich, ethyl silicate coatings.⁽²⁷⁾ A gauze cloth is made into a pad and saturated with a solvent such as methyl ethyl ketone or acetone. Then, using thumb pressure, the solvent-wet pad is rubbed back and forth over the coating. Although the test is said to be imprecise because of variability in thumb pressure from operator to operator, it does provide quick, useful results, particularly on a comparative basis. Other tests can be found to determine solvent resistance.^(28–31)

4.2 Mechanical Properties

Mechanical properties such as tensile strength, elongation, and toughness and related parameters are important characteristics of coatings.⁽³²⁾ Outdoor coatings must withstand the rigors of the tensile and compressive forces that occur and cause expansion and compression during each day and with the changing seasons as the temperature changes. Hail and sleet challenge the toughness of coatings. The effects of temperature on coatings for wood substrates are further complicated by the differences in hardness, chemical composition, and expansion coefficients of spring wood and summer wood. Obviously, these changes occur many, many times over the course of a number of years. Many coatings are applied to a substrate before forming operations are carried out. Consider the metal white caps that are applied to a variety of packaged foodstuffs. The caps are formed after the white coating is applied to sheet steel. The coated metal is subjected to severe tensile and shear forces during the strong bending and twisting operations required to form the cap. The coating–steel composite must have excellent adhesion, strength, and toughness to withstand the torturous operation.⁽³³⁾ Three-piece beverage cans undergo severe bending and twisting when the can ends are combined with the can body in a flanging operation. The same is true



Scheme 1



Scheme 2

when the lid is attached to a two-piece can. In addition, when the filled cans are transported by truck, the coated, filled can is again subjected to twisting and flexing during handling and with each bump and turn in the road during transportation. The severity of these operations is such that pin holes and failure can develop in the container and cause product loss as well as potential liability.

Tensile properties are those characteristics that a material exhibits when a uniaxial force F is applied to a specimen of length L_0 and cross-sectional area A , as depicted in Figure 1. Under no-load conditions, the specimen is at rest. When the force is applied, the specimen experiences a tensile stress, σ , that is equal to the applied force per unit area and an elongation to a final length, L , involving a tensile strain, ϵ , which is given by change in length per unit length or $(L - L_0)/L_0$. In the region where stress is directly proportional to strain, a tensile modulus, E , can be defined as the slope of the stress-strain relationship as shown in Equations (1) and (2). (Similar expressions can be defined for shear and bulk deformation conditions.)

$$E = \frac{\text{Tensile stress}}{\text{Tensile strain}} = \frac{\text{Force per unit area}}{\text{Change in length per unit length}} \quad (1)$$

$$E = \frac{F}{A} \div \frac{\Delta L}{L_0} = \frac{\sigma}{\epsilon} \quad (2)$$

However, polymers and coatings actually have no measurable region where stress is proportional to strain because polymer molecules can flow under an applied force. That is, polymers are not elastic in nature; rather they are viscoelastic and slowly flow when placed under conditions of loading. The modulus of viscoelastic materials has a storage or completely recoverable elastic component (E') and a viscous or loss component (E'') that is not recoverable and the energy of which is lost through viscous heating

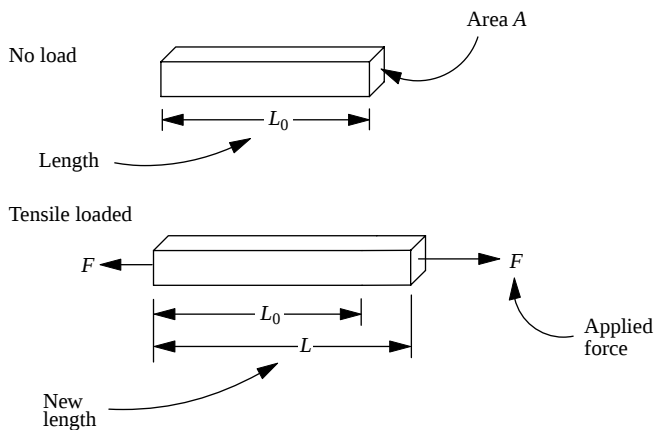


Figure 1 A specimen at rest and in a tensile-loaded condition.

during the stressing process. To circumvent this problem, when measured under static conditions, the value of E is calculated by measuring the stress at some stated, fixed strain, usually 1% or less, and dividing it by the strain. Such a measured value of E is denoted as the secant modulus⁽³⁴⁾ measured at the selected strain.

Dynamic mechanical analysis (DMA) is a technique that enables the two components E' and E'' , which together are known as the complex tensile modulus, E^* , to be distinguished and measured.^(35,36) In DMA, the stress or strain described in Figure 1 is applied to the specimen in an oscillatory manner that is usually described as being sinusoidal in nature, though the exact nature of the deformation depends on the particular instrument used for the measurements. A sample is held under sufficient tension that it is not limp at the lowest applied oscillatory strain. Although the impressed wave motion is the same, say sinusoidal, for the stress and the strain, because of the material's viscoelastic nature these properties are out of phase by an amount or angle, δ , the phase lag. For an ideal elastic material, δ is zero and the stress and strain are in phase. For a Newtonian liquid, if it could be so tested, δ would be 90° . For viscoelastic materials, δ is between 0 and 90° . These components of the modulus can be represented as two vectors that are 90° out of phase with each other, as described in Figure 2.

Equations (3–6) describe the stress wave as two waves, one in phase with the strain (the elastic response) and the other 90° out of phase with the strain (the viscous response).

$$\text{Storage modulus} \quad E' = \frac{\sigma_t \cos \delta}{\epsilon} \quad (3)$$

$$\text{Loss modulus} \quad E'' = \frac{\sigma_t \sin \delta}{\epsilon} \quad (4)$$

$$\text{Complex modulus} \quad E^* = [E'^2 + E''^2]^{1/2} \quad (5)$$

$$\text{Loss tangent} \quad \frac{E''}{E'} = \frac{\sigma_t \sin \delta}{\sigma_t \cos \delta} = \tan \delta \quad (6)$$

The complex tensile modulus, E^* , or the one that is actually sensed, can be calculated from the components, E' and E'' , by the Pythagorean theorem. The ratio of the viscous response to the storage response is equal to the

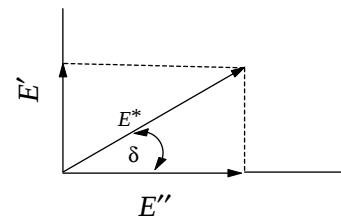


Figure 2 Vector representation of the components, E' and E'' , of the complex shear modulus, E^* , and the phase angle, δ .

tangent of the out-of-phase angle and is known as the loss tangent. When these parameters are measured as a function of temperature, the loss modulus and the loss tangent exhibit maximums at the glass transition temperature, T_g , and at other temperatures if there are other secondary loss mechanisms. Below T_g , materials are glass-like in nature – hard and brittle – and above T_g they are soft and flexible in nature. If the system is cross-linked, above T_g the material will exhibit an extensive rubbery nature.

To visualize the storage and loss components of a material, consider a rubber ball that is dropped from some height onto a hard, elastic surface. The ball will bounce up and down with a decreasing height as each succeeding bounce takes place until it is finally at rest. The elastic nature of the ball causes it to try and return to the release point by means of stored energy, but the viscous nature of the ball results in it losing energy as heat to the surroundings; consequently, the ball reaches a height somewhat less than the original height. The motion is gradually damped until finally the ball ceases to bounce. One readily knows that a markedly different result is obtained if this experiment is performed on a summer day than on a winter day. Actually one can measure important physical properties of materials by this technique, but a number of more practical methods than this one exist.⁽³⁶⁾

4.3 Flexibility and Toughness

Flexibility is the ability of a coating to be bent or flexed in forming operations without cracking, losing adhesion, or failing in some other manner. Toughness is the ability of a coating to withstand large stress forces imposed over a short time without cracking, rupturing, shattering, or tearing.⁽³⁷⁾ Coatings must properly perform during manufacturing operations, during use, and often during misuse. To do this, they must have sufficient flexibility and toughness to withstand failure when subjected to bending and twisting, as is encountered in forming operations, to expansion and shrinking during temperature changes, and to mechanical abuse.

Flexibility is usually measured by a mandrel bend test⁽³³⁾ or a T-bend test.⁽³⁵⁾ The mandrel bend test involves bending a coated substrate, usually sheet metal or rubber-type materials, over either a conical mandrel or over cylindrical mandrels of various diameters. The standard, smooth-steel, conical mandrel has a length of 203 mm (8 in) and a diameter of 3 mm (0.125 in) at one end and 38 mm (1.5 in) at the other end. The coated substrate, coating side up, is bent around the mandrel with a lever device and the extent of cracking, if it exists, is determined. The distance from the small end of the mandrel to the crack is determined visually and can be used graphically to determine the percent elongation.

(However, there is no indication in the test method that elongation determined from tensile studies will yield a value related to the cracking-failure point.) The mandrel diameter at the point where cracking ceases is reported as the resistance to cracking resistance or flexibility.

The cylindrical mandrel test is a pass/fail test that involves placing the coated substrate over a mandrel, coating side up, and bending the specimen about 180° around the mandrel by hand at a uniform velocity in a specified time. Usually six mandrels having diameters ranging from 25 mm (1.0 in) to 3.2 mm (0.125 in) are used. The panel is bent over the largest diameter mandrel and then immediately examined for cracking. If none occurs, the next smaller mandrel is used and so on until failure occurs or the smallest diameter mandrel has been passed. The smallest diameter at which cracking does not occur is reported. The test can be used to calculate coating elongation.

The T-bend test involves placing a coated metal panel with a 50 mm (2 in) minimum width in a smooth jaw bench vise and holding it firmly.⁽³⁸⁾ The panel must be sufficiently long that the needed number of bends can be made, i.e. about 150 mm (6 in). Then the panel is bent 90° with the coating on the outside of the bend, removed, and further bent by hand until the bent end can be inserted in the vise; the vise is tightened to complete the 180° bend. The apex end of the bend should be as flat as possible. This is termed a 0T (zero-T) bend. The bend is then examined with a 5 to 10 power magnifier for cracks and pressure-sensitive tape is applied and removed to determine if coating can be picked off. The process is then repeated by placing the bent end in the vise and bending through 180° around the 0T bend. This forms the 1T bend. This is continued for 2T, 3T, etc. bends. The lowest T bend at which no cracks are visible and there is no pick off of coating is the value reported. Note that the radius of curvature of the bend increases with each succeeding bend and coating elongation required to make the bend decreases with each succeeding bend.

Flexibility of pipeline coatings that are to be subjected to short-radius bends is determined by bending the coated pipe around a designed, variable-radius mandrel to produce a range of short-radius bends.⁽³⁹⁾ Coating failure is apparent by visual and/or electrical inspection of cracking or loss of adhesion.

Toughness can be defined as the ability of a coating to withstand an impact without cracking or breaking. It is dependent on the nature of the polymer or polymers used in the coating and on adhesion. Impact resistance, which is related to formability, can be measured by dropping a weight from various heights through a guide tube onto an indenter that rests on the surface of the coated substrate.⁽⁴⁰⁾ The test can be made on the coated side (face impact) and/or the uncoated side (reverse impact)

of the coated substrate. Cracking or other failure is noted on or around the dimple caused by the indenter. The cited ASTM test gives three procedures for ascertaining failure: visual inspection, application of an acidified copper sulfate solution, and use of a pin hole detector. Several impacts are made at different impact values and at the same impact value. The value where the force required changes the result from mainly passing to failing is the test end-point. The result at this point is reported as kilogram-meters (inch-pounds) impact resistance.

A wedge bend device is used to determine impact resistance and formability of metal strips that have been factory coated by a roll coating or other application technique.⁽⁴¹⁾ Coated strips are bent 170–180° over a 3.2-mm (0.125 in) cylindrical mandrel that is attached to the impact platform. A 1.82-kg (4 lb) guided rod with a flat end is then dropped onto the test specimen. Variation in the height of drop allows the force needed to crack the coating to be measured. A test that involves high-pressure pressing of an indenter ball into a zinc-rich primer-coated metal substrate tests the formability of the coated metal.⁽⁴²⁾ Formability tests that ascertain the flexibility and impact resistance of coatings by stamping a die into coated metal exist.⁽³⁷⁾

The impact resistance of pipeline coatings is determined by a limestone drop test,⁽⁴³⁾ a falling weight test,⁽⁴⁴⁾ and a penetration resistance test.⁽⁴⁵⁾ The limestone drop test involves dropping weighed amounts of a particular type of limestone through a chute onto a coated pipe. The number of drops required to penetrate the coating by either visual or electrical inspection is reported as the impact resistance. The falling weight test is similar to that described above⁽⁴⁰⁾ except special pipe-holding devices and impactor surface characteristics are involved. Coating breaks or penetrations are detected by measurement of electrical resistance changes; the impact resistance is the amount of energy required to cause a break. The penetration resistance test involves applying a blunt rod loaded with a dead weight to a coated steel pipe. The depth or rate of penetration of the rod into the coating is measured as a function of time. This and any failure (cracking or other penetration) are reported.

4.4 Adhesion

The importance of adhesion, the ability of a coating to resist removal from the surface to which it is applied, is self evident.⁽⁴⁶⁾ Such adhesion can be between substrate and coating, between a primer coating and a top coating, between coatings applied to an existing coating, etc. In addition, the coating must adhere under various weathering and cleaning, usually aqueous, conditions. The adhesion can be between the same – in a chemical sense – materials or between a broad variety of materials

including plastics, wood and other cellulose, metals, ceramics, etc. There are two aspects involved in adhesion: “basic” adhesion, which is the combination of all intermolecular and interfacial forces, and “practical” adhesion, which is the work needed to disrupt the adhering combination. Practical adhesion is almost always the quantity measured in the coating industry.

The most common method of testing coating adhesion involves applying an adhesive tape to the coating, which is either uncut or cut in some manner, and then removing the tape under specified conditions. The cut surface is observed and the degree to which the coating is removed is compared against standards. The test is considered simple to perform and low in cost. A widely used test method for coatings on metallic substrates⁽⁴⁷⁾ involves making an X-cut in the film (method A) or making a lattice cut with a device that makes six or eleven cuts in each direction (method B) with each of the cuts made through the film to the substrate. A transparent, pressure-sensitive tape is applied to the cut area and removed in a prescribed manner. The coating is then visually examined and rated on a zero to five comparison scale. On this scale five indicates no removal and zero indicates greater than 65% removal from the scored area, with various descriptions for the values between the extremes. Method A is meant to be used on the job and method B is meant for use in the laboratory. The test and results are qualitative in nature, and the results are considered reproducible within one unit when the substrate is metal. On plastic substrates, reproducibility is poor since the test is not designed for relatively soft substrates that are usually coated with brittle coatings.

Adhesion of coatings to flat substrates can be determined by pushing the panel beneath a rounded stylus on a balance-beam device that is increasingly loaded until the coating is removed.⁽⁴⁸⁾ This scrape adhesion test is used to differentiate the degree of adhesion to substrates. It provides relative rating values for coatings with considerably different degrees of adhesion.

The pull-off strength⁽⁴⁹⁾ or adhesion of a coating is measured by applying an increasing tensile force perpendicular to the coating surface until a plug of material is detached. Alternatively, this test can be a pass or fail test if a prescribed stress is applied and it is determined if the surface remains intact under this stress. The tests are carried out with a portable device with a loading fixture that is secured to the coating surface with an adhesive. The adhesive is either a two-part epoxide or acrylic system. The fixture is aligned normal to the surface, and the tensile stress is applied in a slow (less than 1 MPa s⁻¹, 150 psi s⁻¹), continuous manner until a plug of material is removed. The force attained at failure or at maximum force applied is reported. In addition, the plug is examined to determine the percentage adhesive and

cohesive failures, and the interfaces and layers involved in failure are reported.

4.5 Hardness

Coating hardness is the ability to resist permanent indentation, scratching, cutting, and penetration by a hard object.⁽⁵⁰⁾ Different methods of evaluating hardness yield different results because they measure different qualities of the material. There is no absolute scale and each method has its own scale of defined hardness.

Determining hardness by gouging or scratching the coating with drawing leads or wood pencils of different hardness (from 6B to 6H) is simple and inexpensive; it is widely used in laboratory development work and production control testing.⁽⁵¹⁾ To conduct the test, the pencil is sharpened with a draftsman-type sharpener. The sharpened lead point is then held at a 90° angle to horizontal on No. 400 grit abrasive paper and rubbed until a smooth, flat, circular cross-section is obtained. To carry out the test, the coated panel is firmly held on a level surface and the hardest, sharpened pencil is held on the coating at a 45° angle. The pencil is then pushed away from the operator while using sufficient downward pressure to either cut through (gouge) or scratch the film or to crumple the edge of the lead. This procedure is repeated with softer and softer leads until a pencil is found that will not cut through or scratch the coating. The gouge hardness is reported as the hardest pencil that will leave the coating uncut for a push stroke of at least 3 mm (0.125 in). The scratch hardness is reported as the hardest pencil that will not scratch the coating. Because of the nature of this test, it is operator dependent and results may vary between different operators and laboratories.

Indentation hardness of coatings is determined with sophisticated devices that determine the resistance to penetration by an indenter.⁽⁵²⁾ Knoop indentation hardness (method A) is determined by bringing a pyramidal diamond indenter into contact with the coating and then applying a selected load to the indenter and maintaining the load for 18 ± 0.5 s. After this time, the indenter is withdrawn. The Knoop device is equipped with a microscope that has a movable micrometer stage; immediately after the indenter is withdrawn, the microscope is adjusted and focused so that the indentation is in the field of the microscope. With the indentation sharply focused, the length of the long indentation diagonal is determined. The indentation length is converted into Knoop hardness numbers (KHN) with tables supplied by the instrument manufacturer. If the tables are not available, KHN hardness numbers can be calculated.

Pfund indentation hardness (method B) is determined with a device equipped with a microscope that will apply a 1.0-kg (2.2 lb) load to a hemispherical (3.18 mm, 0.125 in

radius) transparent quartz or sapphire indenter that is in contact with the coating surface. The load is applied for 60 s; after that time, with the loaded indenter still in place, the diameter of the circular impression is rapidly measured. An instrument constant (1.27) is then divided by the square of the indentation diameter in millimeters to obtain the Pfund hardness numbers (PHN).

Numerous other methods exist that determine hardness by scraping and indenting as well as by marring and abrasion.⁽⁵⁰⁾ In addition, hardness can be measured with pendulum damping devices⁽⁵³⁾ and rocker devices.⁽⁵⁴⁾ The three methods described above are in common usage.

4.6 Abrasion

Abrasion resistance is the ability of a coating to resist having its original appearance and structure altered when it is subjected to the influence of erosion, rubbing, scraping, or other ablative action.⁽⁵⁵⁾ Both temperature and environment can have an effect on abrasion resistance, but the relationship between these factors and interrelated mechanical properties is not simple. For example, hardness and modulus increase with decreasing temperature, and this may be detrimental to abrasion resistance if the coating loses flexibility or toughness. Increases in humidity around an object or subjecting an object to a moist environment as in washing a wall can soften a coating and alter its resistance to abrasion. Other factors that can have an effect on abrasion resistance include toughness, strength, and other mechanical properties. Because of the complex interrelationship between characteristics that affect abrasion resistance, it is important that the test method subjects test specimens to conditions that are similar to those encountered in actual use.

Many, if not almost all, coated items are subjected to some sort of ablative action, and such action can cause marring and/or wear. These items include appliances, automobiles and other transportation equipment, beverage cans, business machines, farm equipment, floors, furniture, highways (traffic paints), interior and exterior structural walls, and so on. The actions include: wind, rain, hail, and other natural periodic actions; wear that can be relatively continuous, such as automobile traffic or walking; polishing or other rubbing of furniture or an automobile with a harsh fabric; or accidental contact of a coated surface with a button, a toy, or a similar object. An example of rubbing effects caused by similar objects rubbing against each other are those that are encountered when beverage cans rub against each other in a multipack during shipping and handling.⁽⁵⁶⁾

Abrasion resistance of coatings applied to flat, rigid surfaces can be measured by rotating the coating against an abrasive-filled, weighted wheel.⁽⁵⁷⁾ The results are reported as the number of cycles to remove a unit amount

of coating (wear cycles per 25.4 μm), as the loss in weight per cycle multiplied by 1000 (wear index), or as the weight loss determined at a specific number of cycles (weight loss). Although this test method is fairly simple to carry out, reproducibility is poor. It is recommended that results be limited to testing in only one laboratory when numerical values are to be used. Agreement of results between laboratories is markedly improved if different coatings are merely ranked rather than trying to compare numerical values.

Coatings on non-planar surfaces such as those found on pipelines are tested for abrasion resistance by placing the externally coated pipe through a specially designed steel drum apparatus and eroding it with an aqueous, abrasive slurry contained in the horizontally revolving drum.⁽⁵⁸⁾ The specimens are electrically insulated from contact with the drum. The test is applicable to all types of electrical insulating coating including thermoplastic and thermoset coatings and bituminous materials. Measurement of electrical resistance changes between the pipe and the drum as the coating erodes indicates the coating abrasion resistance. Because of electrical requirements, metallic protective coatings such as zinc are not tested by this method. However, such coatings and others designed to function as electrical barriers are tested for cathodic disbonding by other tests.^(59,60)

Painted interior house walls are soiled near doorways, windows, play areas, cooking areas, etc. Such soiled areas and often the entire walls are cleaned by repeated scrubbing, and during the scrubbing the paint is subjected to corrosion. The relative erosion resistance of interior, flat wall paints to wet scrubbing can be determined by applying the paint to particular black plastic panels and scrubbing with a nylon bristle brush that is wet with an aqueous, detergent-based scrub medium.⁽⁶¹⁾ The wet brush is driven in one direction across the coated surface; after each set of 400 cycles the brush is removed, the scrub medium is replenished, and the brush is replaced. This procedure is repeated until the paint film has been removed. The number of cycles to failure is reported. The test is designed for freshly painted surfaces rather than aged surfaces. The degree of erosion of exterior paints, which occurs mainly by chalking, can be determined by comparison of the surface with pictorial standards.⁽⁶²⁾ Pictorial comparisons are also used to evaluate the wear resistance of traffic paints.⁽⁶³⁾

Abrasion resistance is also determined by air-blasting silicon carbide grains at the coated panel at a 45 g min^{-1} flow rate.⁽⁶⁴⁾ The abating is continued until the coating is worn through. At this point, the blasting is terminated, and the amount of ablative used is determined. The abrasion resistance is determined as the grams of ablative used per 25.4- μm film thickness. A similar test involves dropping a silica or silicon carbide abrasive through a

guide tube from a specified height onto a coated planar surface.⁽⁶⁵⁾ Silica (sand) is a milder abrasive than silicon carbide and the slower rate of abrasion it causes can be useful in discriminating between different coatings. Basically this test differs from the previous test in that the ablative contacts the coating under a gravity flow rate rather than an air-forced flow rate. The abrasion resistance is determined as the kilograms of ablative used per 25.4- μm film thickness.

4.7 Slip

Usually slip,⁽⁶⁶⁾ meaning the opposite of traction or clinging, is not an inherent property of coatings unless one is discussing the tetrafluoroethylene polymers and copolymers. Coatings are said to have good slip when they have a low coefficient of friction and poor slip when they have a high coefficient of friction. Slip indicates the ease with which two contacting surfaces can move by each other. Coatings are said to have slip when they have a tack-free surface and behave as if they were lubricated.

Slip is an important characteristic of coated objects for it is the property that allows coated materials to slide by one another in forming operations, during filling, handling and shipping, as well as in other manufacturing and use operations. However, it is worth pointing out that the surface can be too slippery, that is have too low a coefficient of friction. Imagine a beverage can with a surface so slippery that it could not easily be held in a person's hand. Also, a low coefficient of friction can be an undesirable characteristic in floor coatings, since people walking on the surface could slip and fall or vehicles could slide and cause damage or harm. Gymnasium floors, porch and deck floors, concrete work-area floors, and kitchen floors are areas where this is of particular concern.

Slip can be imparted to films by incorporating a compound into a coating formulation that is incompatible with the dried or cured coating; it will then exude to the surface of the coating. A way of imparting slip to flat, coated metal sheets is to spray lightly a very low-volatility lubricant onto the coating just after the coating is cured and prior to stacking for the next manufacturing operation. Compounds such as wax esters, fatty esters, alkanolamides, metallic stearates, waxes, and silicones are used to decrease frictional resistance or to control slip.

Slip is determined by measuring the frictional properties of coatings. Friction is the force between surfaces that opposes imposed sliding motion. It is the characteristic that determines the resistance to slip or the magnitude of slip.

In one method,⁽⁶⁷⁾ the static friction of coatings is determined by an inclined plane sliding test or a horizontal pull test. The inclined plane test employs one or more weighted sleds that are individually placed on the coated

surface, which is fixed to a flat, movable surface. The movable surface is then inclined from the horizontal at a rate of $1.5 \pm 0.5^\circ \text{ s}^{-1}$ until the sled begins to slide down the inclined coating surface. The tangent of the angle of inclination at this point is reported as the static friction. The horizontal pull test has a weighted sled placed on a specimen that is fixed to a flat, horizontal base. The sled is then pulled across the specimen with a mechanical power unit, and the force required to start the sled moving is determined. This force divided by the mass of the sled is reported as the static friction. Static friction determined by this method is useful for ascertaining the slipperiness of floor polishes, the slip resistance of footwear on floor tiles and floor coatings, the appropriateness of coatings for the exterior of cans, etc. The measurements are also useful to determine the effect of coating additives or spray lubricants on the slipperiness of coatings. A number of methods for determining friction can be found in the literature.⁽⁶⁶⁾

4.8 Stress in Coatings

Stresses can develop within coatings during film formation, through temperature changes, and through relative humidity (RH) changes.⁽¹⁵⁾ These internal stresses have an effect on coating degradation. They affect adhesion and/or cohesion and have an effect on delamination and cracking. Thermoset coatings have higher internal stresses than coatings that do not involve cross-linking compounds, such as lacquers and alkyds.

Although internal stresses can have a detrimental effect on adhesion, they originate through the process of adhesion. This seeming paradox can be readily understood if the following is considered. To protect a substrate adequately, good adhesion between the substrate and coating is required. However, adhesion causes immobility of the coating at and near this interfacial area, which, in turn, does not allow the coating to move in a normal manner, for example when the temperature changes.

When a solid coating film forms, a liquid is changed into a solid. While the film is liquid, the coating is mobile and volume contraction can take place with no stress development. As a solid coating film forms, in almost every instance contraction continues to take place but is restricted by adhesion. As a result of this restriction, tensile stresses develop within the coating. However, as soon as stress develops, the molecules seek to relieve the stress and a relaxation process begins. Therefore, as film development continues, stresses within the film can increase, decrease, or remain constant depending on the rate of stress development and of stress relaxation. It should be noted that stress development begins when the T_g of the changing system is reached. In the case of a

coating that is formed from a solution of polymer, this is at the point where the T_g of the solvent/polymer solution is equal to the experimental temperature.

If it is assumed that the internal stress is in a plane parallel to the substrate and is isotropic in nature, the internal strain ε_i can be described by Equation (7):

$$\varepsilon_i = \frac{V_s - V_t}{3V_s} \quad (7)$$

where V_s is the coating volume at the solidification point and V_t is the coating volume at time t after solidification. It is readily apparent that, as the volume decreases as a function of time during final film formation, the internal strain and, therefore, the internal stress increase.

Changes in temperature will cause the dimensions of a coating/substrate combination to change. Since the expansion coefficients of the coating, $\alpha_{c,T}$, and the substrate, $\alpha_{s,T}$, are almost always different, an internal strain, ε_T , is set up. This is described by Equation (8).

$$\varepsilon_T = (\alpha_{c,T} - \alpha_{s,T}) \Delta T \quad (8)$$

Since absorption and desorption of water can cause similar changes in dimensions of the coating, $\alpha_{c,RH}$, and substrate, $\alpha_{s,RH}$, Equation (9) similarly expresses the internal strain that is caused by RH changes.

$$\varepsilon_{RH} = (\alpha_{c,RH} - \alpha_{s,RH}) \Delta RH \quad (9)$$

These stresses act together and may augment each other and be very important or they may negate each other and be small and relatively unimportant (Equation 10).

$$\sigma_{\text{total}} = \sigma_i \pm \sigma_T \pm \sigma_{RH} \quad (10)$$

The component σ_i is always positive, but the contributions from temperature and RH effects can be positive or negative. Positive effects occur in coatings that tend to contract and set up internal tensile stresses. Negative effects occur in coatings that tend to expand and set up compressive stresses. A dry, cold winter day will involve low temperatures and low RH, with high resultant internal tensile stresses. Conversely, a humid, summer day will involve high temperatures and RH, with resultant high internal compressive stresses.

There are a number of ways⁽¹⁵⁾ to measure internal stresses, including brittle lacquer materials, cantilever beams, optical, strain gauges, and X-ray diffraction. The cantilever beam method is most widely used and gives suitable measurements. This method depends on the fact that a coating under stress on a substrate will deflect in the direction that will relieve the stress. There are two types of cantilever beam used. A one-side coated substrate is either fixed at one end or is freely supported on two knife edges. The deflection in either case can be measured;

if the elastic properties of the substrate are known, the internal stress can be calculated.

4.9 Chemical Resistance

Coatings are the first-line defense for a product that contacts hostile environments. They protect many products from a variety of chemicals.⁽²⁸⁾ In addition to protecting the product, it is preferable that the coating does not stain, does not lose adhesion, does not lose gloss, and is not permanently altered in any way by its contact with the hostile conditions.

Household chemicals include alkaline and acid solutions, beverages, condiments, cosmetics, edible and inedible oils and greases, ethyl alcohol, fruit juices, hot and cold water, soap and detergent solutions, vinegar, as well as many other common compounds. These compounds can be placed on coatings and either left open to the air or covered by a watch-glass to determine the effect of the compounds on adhesion, blistering, gloss, softening, and other properties of the coating.⁽²⁹⁾ Furniture finishes are tested for resistance to alcohol, boiling water, cosmetics, hot coffee, and other chemicals.⁽⁶⁸⁾ Each compound is examined in a particular manner. For example, hot coffee is poured onto a coated panel held in a horizontal position and allowed to dry. The coating is then examined for spotting, softening, graying, staining, or any other deterioration. Cosmetics are applied to the coating and placed overnight in a 50°C oven. The coating is then examined for film failure and discoloration.

Various coatings used in the transportation industry are tested in a somewhat similar manner.⁽⁶⁹⁾ However, in some instances, the test includes exposure to either sunlight or ultraviolet light for a specified time; increased temperature is an important additional variable if hot, sunny climates are being simulated. Some of the chemicals important to transportation coatings are alcoholic windshield-washing solutions, antifreeze compounds, gasoline, hydraulic fluids, lubricating greases and oils, polishes, and road oils and tars. An immersion technique⁽⁷⁰⁾ is used to determine the solvent and fuel resistance of traffic paint. Important factors are adhesion loss, blistering, softening, and wrinkling. Other immersion techniques exist for examining chemical resistance.⁽²⁸⁾

Resistance to moisture and water is determined in accelerated cabinets that are intended to determine long-term durability. However immersion in warm water (37.7°C) can be used for comparison purposes.⁽⁷¹⁾ Resistance to aqueous saline fogs is particularly important for aircraft, automobile, and marine coatings or for coatings that are used near oceans or in salty road conditions.⁽⁷²⁾ In this test, X-scored coated metal panels are placed in a cabinet and exposed to a condensing fog

of an aqueous sodium chloride solution. The panels are periodically examined to determine the degree of rusting. Details for nonmandatory construction of a suitable cabinet are given in the test method.

5 END-USES

Earlier discussions of coating uses indicated the wide variety of end-uses. Testing of coatings can be fundamental and sophisticated in nature or it can be quick and simple to accomplish. Testing may be done to meet a set of specifications set by the supplier and by the seller or user or it may be generalized to meet what a company or group feels is important to a number of end-uses. Simple tests include the pencil hardness test,⁽⁵¹⁾ rubbing tests for solvent resistance (rubbing a coating with a solvent such as acetone, methyl ethyl ketone, or xylene until it fails or passes a given number), or merely scratching a nickel coin across the coating while applying pressure. These simple tests tell one skilled in the art if the coating will be hard or soft, if it will be solvent resistant, and if it is tough and adherent while having mar resistance. Combinations of such test are useful to the in-house experts who wish to test coatings as they are produced. Each expert readily knows if the tests or others they have devised will mean that the coating being manufactured will meet requirements. However, particular end-uses require the use of specific tests, some of which were mentioned above. Others are developed to meet the requirements of specific industries.

5.1 Tests Required for Specific End-uses

Although the specific tests described below are referenced, often a buyer and a seller will define certain requirements that must be met by tests such as these or those that will define mutually acceptable testing criteria.

Aerospace and aircraft coatings⁽⁷³⁾ are tested for adhesion under ambient conditions and under specific environments, such as after water immersion with the Scotch tape peel test.⁽⁴⁷⁾ Scrape adhesion⁽⁴⁸⁾ is also important. Flexibility is determined by the mandrel bend test⁽³³⁾ at temperatures as low as -51°C. Toughness is determined by measuring impact resistance with the falling weight test (see above)⁽⁴⁰⁾ and/or with a G. W. Impact-flexibility Tester.⁽⁷⁴⁾ The latter test involves dropping a steel cylinder that has spherical knobs on its surface onto a coated panel. The knobs will subject the coating to elongations of 0.5–60% under the conditions of the test. Other important mechanical properties include hardness and mar resistance.^(48,51) Tensile properties are determined with free films.

The aluminum and steel beverage container industry⁽⁵⁶⁾ is a large consumer of coatings and requires coatings to have high-quality mechanical characteristics in addition to meeting governmental regulations for safety and health. The inside and the outside of the cans are coated but with different coatings. The inside coating provides protection of the metal can from its contents as well as protection of the contents from taste alteration or contamination by contact with the metal can. The outside coating provides attractive, product identification. Important mechanical properties include abrasion resistance,⁽⁵⁷⁾ adhesion,⁽⁴⁷⁾ hardness,⁽⁵¹⁾ and flexibility.^(38,40)

Pipelines carry oil, natural gas, water, and chemicals to plants and to consumers. Often the product is transported for long distances and often these pipes are buried underground and, thus, have a constant pressure applied. During the burial process, dirt, stones, and rocks are thrown into the trench and the coatings must have sufficient toughness to withstand this rough handling. Chemical plants and refineries use extensive pipeline systems to carry raw materials to reactors, intermediates to separation or other reaction systems, and final products to shipping facilities. The coatings must have sufficient integrity to withstand the hostile environments associated with such use. Above- and below-ground pipes are subjected to expansions and contractions as the temperature changes; again toughness and flexibility are important factors. Although pipelines are efficient means to carry out such operations, they must be protected by coatings to ensure dependable service and long life.⁽⁷⁵⁾

Automotive coatings⁽⁷⁶⁾ are subjected to mechanical abuse from ordinary usage and from nature. Although individual automobile manufacturers have specific testing protocols involving test methods devised in-house, they often use many of the tests described above. A variety of substrates are involved and range from those that are flexible to those that are rigid; the coatings range from primers, to guide coats, to topcoats, etc. Hardness is determined by the Tukon indentation method⁽⁵²⁾ and wear resistance by the Taber Abraser method.⁽⁵⁷⁾ Adhesion is determined by either X-scribing or cross-hatching and applying pressure-sensitive tape under ambient conditions⁽⁴⁷⁾ and after exposure to 100% RH.⁽⁷⁷⁾ Coatings must have at least 99% adhesion when tested by these methods. To test resistance to impact from stones and road debris, a gravelometer test is used.⁽⁷⁸⁾ Toughness is ascertained by scraping coatings on rigid substrates with a dime and by scraping with a knife when the coating is on flexible substrates. When these scraping tests are used, the coating should not flake, peel, or lose adhesion. Resistance to water⁽⁷¹⁾ and to saline solutions are important aspects of transportation coatings.

Testing of coatings is usually carried out before the coating is chosen for an end-use in order to assess when or how the coating will fail, if it does, when in use. Many coatings are used to protect substrates and thus preserve materials and conserve natural resources; as such, coatings are environmentally sound in nature. Although they can be well designed, coatings can and do fail. Therefore, it is also important to investigate coatings when they have failed to determine the reasons for failure. Mills⁽⁷⁹⁾ has given an excellent description of coating failure analysis.

ABBREVIATIONS AND ACRONYMS

ASTM	American Society for Testing and Materials
DMA	Dynamic Mechanical Analysis
KHN	Knoop Hardness Numbers
PHN	Pfund Hardness Numbers
RH	Relative Humidity

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